

THERMAL ANALYSIS OF SULPHUR IMPREGNATED ACTIVATED CARBONS WITH MERCURY ADSORBED FROM THE VAPOUR PHASE

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Activated carbons (AC), particularly those containing sulphur, are effective adsorbents for mercury (Hg) vapour at elevated temperatures. Activated carbon-based technologies are expected to become a major part of the strategy for controlling mercury emission from coal-fired power plants. Understanding the mechanism of mercury adsorption on sulphur impregnated activated carbons (SIAC) is essential to optimizing activated carbons for better mercury removal efficiency and to developing technologies for the handling of the spent AC.

In this work thermal analysis before and after mercury uptake was carried out for the SIAC prepared under various conditions from oil-sand petroleum coke using a simultaneous differential thermal analyzer. Samples were heated at 20°C min⁻¹ under nitrogen in the temperature range from ambient to 1000°C. The DSC curves suggest both endothermic and exothermic changes during heating. The endothermic processes were attributed to evaporation of moisture and other volatile components. The exothermic processes existed in a wide temperature range of 150–850°C likely due to the oxidation reactions between carbon and adsorbed oxygen, oxygen-containing surface groups. The enthalpies of liquid mercury interaction with SIAC at different Hg/AC mass ratio were also measured at 30, 40 and 50°C using a differential scanning calorimeter. The combination of thermal analysis and calorimetry techniques enabled confirmation that the interaction of mercury with SIAC involves both physical and chemical processes.

Keywords: calorimetry, mercury, sulphur impregnated activated carbons, thermal analysis

Introduction

Mercury is a toxic substance and considered as one of the most harmful metals found in the environment. Its release to the environment must be limited. Since mercury is usually present in low concentration, adsorption processes are most suitable for its removal, for which activated carbons are widely used. Activated carbon can also be used as a carrier for various impregnates which chemically react with mercury and improve mercury removal efficiencies. There are many carbon-containing materials which are used for AC production such as coal, wood, coconut-shell charcoal [1], sago waste [2], coir-pith [3], rubber of tire wastes [4], peat, and petroleum residues [5–7].

Activated carbons are known as adsorbents for various inorganic and organic substances with high adsorptive capacities, e.g. oxygen, carbon dioxide, xenon, and arsenic from wastewaters [6], precious metals (silver and gold) from multi-component solutions with high selective extraction [7], aromatic hydrocarbons (benzene, toluene, ethylbenzene and *p*-xylene) [6, 8], for air purification in food and vegetable warehouses [6], and for radioactive rare gases formed during the operation of nuclear power plants [5, 6].

The sulphur impregnated activated carbons (SIAC) are effective adsorbents for Hg adsorption from

water [9] and vapour phase [10, 11]. It was found that oil-sand fluid coke can be activated by sulphur dioxide and used as an effective adsorbent for removing mercury from gaseous and aqueous media such as flue gases, industrial waste and oil-sand waste and for controlling Hg emission from coal-fired power plants [12]. Thermal analysis and calorimetry are very important techniques for materials characterization and study of different processes. Therefore, many authors have used thermal analysis and calorimetry together with other techniques to describe various processes and materials including activated carbons [13–18].

The purpose of this work is to study the interaction between mercury and sulphurized activated carbons prepared from petroleum coke under different conditions. Thermal analysis before and after Hg uptake was carried out and the enthalpies of liquid mercury interaction with SIAC were measured at various Hg/AC mass ratio at 30, 40 and 50°C.

Experimental

Thermal analysis of SIACs was carried out before and after Hg uptake using a simultaneous differential thermal analyzer, SDT Q600 (TA Instruments). Measurements

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Table 1 Characteristics of activated carbons used in differential thermal analysis

Type of AC	BET SSA/m ² g ⁻¹	Sulphur/%
SIAC-commercial	660	8.1
SIAC-5*	1170	5.1
SIAC-11**	50	11
Virgin activated carbon	800	0.0

*SIAC-5 was chemically activated with KOH at 700°C for 1 h and subsequently treated with a mixture of SO₂ and N₂

**SIAC-11 was chemically activated using a mixture of KOH and sulfur species

were conducted under nitrogen gas (purity 99.9999%) at a flow rate of 100 mL min⁻¹. Samples in the amount of 10–15 mg were placed in a platinum crucible and heated at a linear heating rate of 20°C min⁻¹ over a temperature range from ambient to 1000°C.

The activated carbons were prepared from oil-sand petroleum coke or purchased (Barrick). Types and characteristics of the activated carbons used in thermal analysis are presented in Table 1. The total sulphur content in SIAC was determined by Eschka method based on ASTM standard (ASTM D3177). The specific surface area (SSA) and pore size distribution were determined with the BET-surface area analyzer (Beckman Coulter SA3100). The replicate thermal analysis made for SIAC commercial showed good convergence: the standard deviation did not exceed ±0.8% in mass loss and ±1.94°C in temperatures for ‘peaks’.

X-ray absorption near edge spectroscopy (XANES) analysis was applied to determine the sulphur forms in SIAC-11 and SIAC-commercial. The pyrites and sulphates were observed in SIAC-11 as the major forms of sulphur. The major forms of sulphur in SIAC-commercial were elemental sulphur and sulphates. Species of sulphur in SIAC were identified using XANES at the Synchrotron Radiation Center (SRC) situated on the 1G eV electron storage ring at Aladdin, University of Wisconsin (USA). Both the total electron yield (TEY) and the fluorescence yield (FY) spectra at the sulphur K-edge were recorded and analyzed with Athena software.

Enthalpies of liquid mercury interaction with activated carbons were measured directly by using a differential scanning calorimeter (DSC C80, Setaram). Commercial mixing stainless steel cell (Setaram) was used for the enthalpy measurement. The experimental

cell held two glass ampoules of different size; the smaller ampoule containing a known amount of AC was inserted into a bigger one which contained a known amount of liquid mercury. The reference cell contained only AC. Liquid Hg and AC were mixed by breaking the smaller ampoule with a stainless steel needle when the temperature of calorimeter was stabilized after 2–3 h. Enthalpy of the mercury – AC interaction was measured at different Hg/AC mass ratios (0.25 to 3.00) under isothermal conditions at 30, 40 and 50°C. The amount of AC was fixed at 0.0400 g while the mass of liquid mercury (purity 99.9999%, electronic grade; Sigma-Aldrich) was varied. The activated carbons were dried in an oven at 105°C for 1 h, then cooled down in a desiccator and used in the enthalpy measurements. The total heat of Hg–AC interaction was evaluated by integrating the heat flow curves recorded. The replicate measurements of change in enthalpy of Hg–AC interaction showed good reproducibility with maximum deviation of ±2%. The activation conditions and properties of activated carbons used in calorimetric study are presented in Table 2.

The performance of the DSC was first evaluated by measuring the enthalpy of dissolution of potassium chloride (Standard Reference Material 1655, the National Institute of Standards and Technology) in water at 25°C as a reference test for the calorimeters [19–21]. The experimental enthalpy of dissolution of KCl, 17.25±0.01 kJ mol⁻¹ was in very good agreement with the literature value of 17.22 kJ mol⁻¹ [22].

Results and discussion

Differential thermal analysis

Three samples (SIAC-5, SIAC-11 and SIAC commercial) were subjected to thermal analysis before and after Hg adsorption. The SIACs were used in the mercury vapour adsorption experiments conducted at 75°C. Results of mercury vapour uptake are summarized in Table 3.

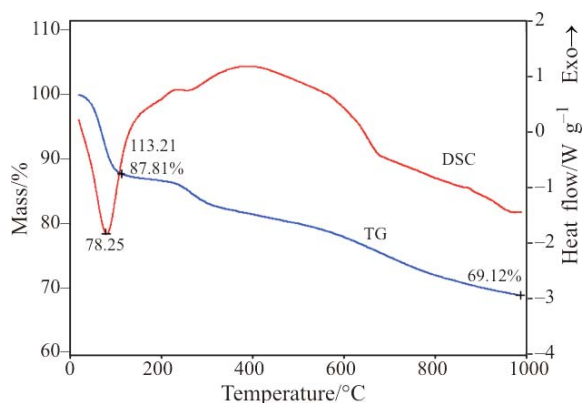
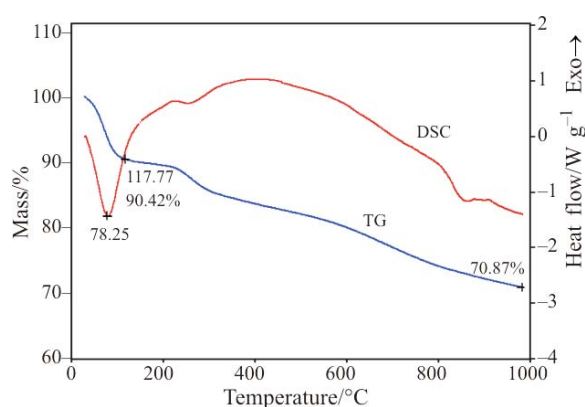
The DSC curve for SIAC-5 before Hg adsorption (Fig. 1) showed both exothermic and endothermic processes. The large endothermic peak at temperatures below 150°C was accompanied by a substantial amount of mass loss of ~12%. This peak is attributed to the loss of moisture via evaporation. Tang *et al.* reported

Table 2 Properties of activated carbons used in calorimetric study

Type of AC	BET SSA/m ² g ⁻¹	Mesopore (t-plot) SA/m ² g ⁻¹	Micropore SA/m ² g ⁻¹	Sulphur/%
SIAC-SO ₂	40	27	14	10.2
AC-KOH	1930	1560	370	<1.0
SIAC commercial	660	430	240	8.1

Table 3 Performance of SIAC in mercury vapour uptake at 75°C

Type of AC	AC/mg	Hg uptake/ μg	Hg in spent AC/%
SIAC-commercial	140	2.1	0.002
SIAC-5	145	36.0	0.025
SIAC-11	109	41.6	0.038

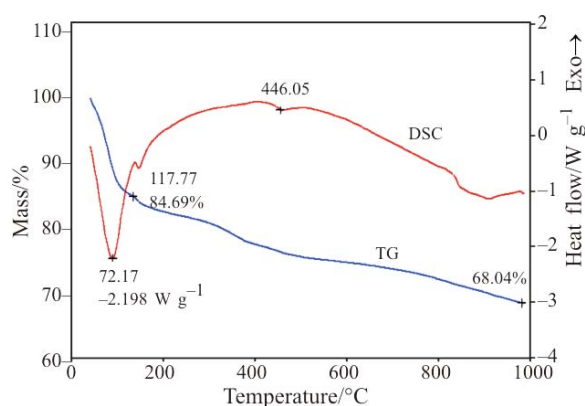

Fig. 1 DSC and TG curves of SIAC-5 before Hg adsorption

Fig. 2 DSC and TG curves of SIAC-5 after Hg adsorption

the removal of the adsorbed water molecules from carbon surface during heating at temperatures up to 150°C [23]. The endothermic peak was followed by a big, but less sharp exothermic 'peak' in a wide temperature range of 150–650°C, accompanied by gradually mass loss. Within the big exothermic peak (Fig. 1) there was a small endothermic peak at temperatures between 230 and 270°C, which was accompanied by an accelerated mass loss of 2%. Ma *et al.* also reported that the heating of Victorian brown coals in inert (nitrogen) atmosphere was accompanied by exo-effects and gradually mass reducing in the temperature range of 150–700°C [24]. The thermal decomposition of Australian coals [25] and Seguruk asphaltite [26] heated under nitrogen atmosphere up to 950°C was also accompanied by mass reducing attributed to volatile matter releasing (gasification). Since the heating of SIAC samples was carried out under high-purity nitrogen, the wide exothermic peak in the temperature range of 150–650°C was attributed to the outgassing of carbon oxides (CO and CO₂) as a result of thermal decomposition of oxygen-containing surface functional groups. Most likely, during heating the oxygen remains bonded to carbon atoms and desorbed from the surface as CO and CO₂. Tremblay *et al.* have found that during heating of AC, carbon oxides were formed, likely as products of thermal decomposition of oxygen complexes [27]. Li *et al.* [28] identified various oxygen functional groups, including carboxyl, lactone, phenol, and carbonyl, on the carbon surface by means of acid-base titration and temperature-programmed desorption (TPD). The presence of oxygen on carbon surface along with sulphur and other functional

groups and their influence on Hg adsorption were discussed by others [28, 29].

Figure 2 is for the same SIAC-5 but after Hg adsorption. Apparently, data depicted on Figs 1 and 2 are quite similar at temperatures up to ~700°C. The only difference is a small endothermic peak at temperatures around 850°C. However, the endothermic peak was not associated with additional mass loss, suggesting a phase change of condensed phases. Although the peak may be linked to some Hg-containing species, there is no other supporting evidence. The overall mass losses are quite compatible as well, 30.9% (Fig. 1) and 29.1% (Fig. 2). The amount of adsorbed Hg in SIAC-5 is about 0.02% (Table 3), too small for thermal analysis. These results suggest that Hg adsorption at a moderate temperature of 75°C did not significantly change the thermal behaviour of SIAC-5.

The results for SIAC-11 before and after Hg adsorption are shown in Figs 3 and 4, respectively.


Fig. 3 DSC and TG curves of SIAC-11 before Hg adsorption

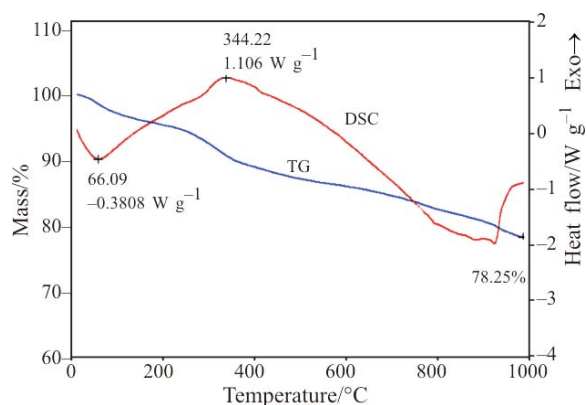


Fig. 4 DSC and TG curves of SIAC-11 after Hg adsorption

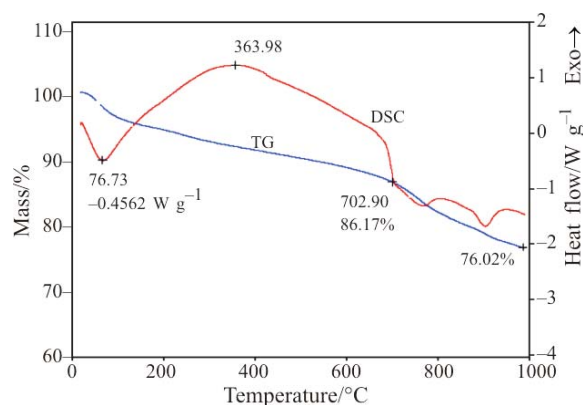


Fig. 5 DSC and TG curves of virgin activated carbon after Hg adsorption

The DSC curves on Fig. 3 show both exothermic and endothermic processes. The endothermic peak below 150°C is attributed to the evaporation of moisture. There is a small endothermic peak at temperatures around 130°C, which is associated with an accelerated mass loss. It is likely due to the evaporation of some volatile component. Within the big exothermic peak (Fig. 3) there was a small endothermic peak at temperatures around 446°C accompanied by a mass loss. This endothermic peak was unique for SIAC-11. Since the major form of sulphur in SIAC-11 is pyrite (FeS₂) it is reasonable to attribute the peak at 446°C to evaporation of elemental sulphur, with a boiling point of 444.6°C, as a result of the thermal decomposition of pyrite in inert atmosphere [30, 31]. The releasing of elemental sulphur from sulphur impregnated activated carbon fibers during heating was also reported by Feng *et al.* [32].

A similar exothermic ‘peak’ accompanied by mass loss (Figs 3 and 4) was observed for SIAC-11. A comparison between Figs 3 and 4 indicates that during Hg adsorption at 75°C about 10% of moisture was removed from SIAC-11, implying that moisture in SIAC-11 is more loosely bound than that in SIAC-5 (Figs 1 and 2). The total mass reduction before and after Hg adsorption was 32 and 22%, respectively (Figs 3 and 4). The loosely bound moisture in SIAC-11 was also confirmed by a substantial amount of moisture removing from SIAC-11 after drying (Fig. 9). Interestingly, the small endothermic peak at about 130°C disappeared after Hg adsorption (Fig. 4), and presented on the DSC curve for SIAC-11 after drying (Fig. 9), implying that some volatile component was not removed by drying at 105°C and likely chemically reacts with mercury at 75°C. The exothermic process was intensified at temperatures over 900°C (Figs 3 and 4), which was also found for sulphur-free activated carbon (Fig. 5), and the process is likely linked to carbon instead of sulphur.

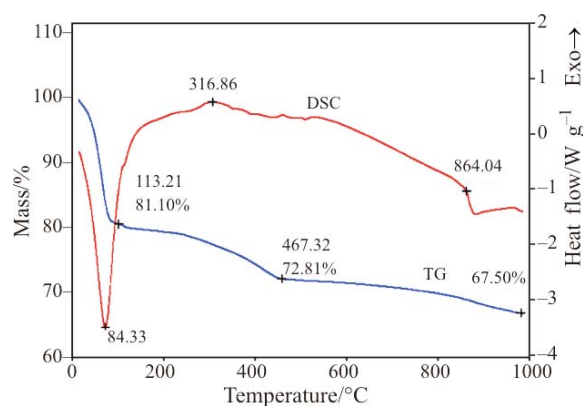


Fig. 6 DSC and TG curves of SIAC-commercial before Hg adsorption

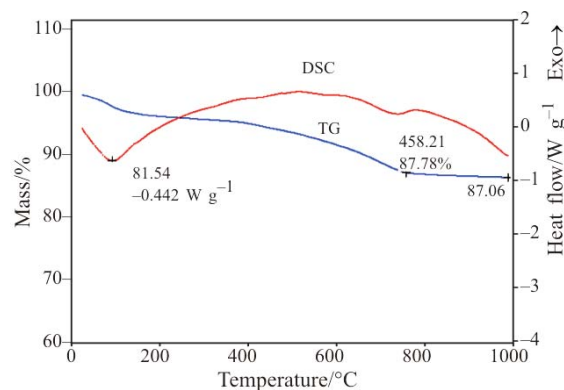


Fig. 7 DSC and TG curves of SIAC-commercial after Hg adsorption

The results for SIAC-commercial before and after Hg uptake are presented on Figs 6 and 7. The thermal behaviour of SIAC-commercial is similar to that of SIAC-5, except greater moisture content. As shown in Fig. 7, the Hg adsorption experiment at 75°C was able to remove most of the moisture, suggesting the water was loosely bound in SIAC-commercial as it was also indicated for SIAC-11. A small

Table 4 Mass loss (%) by SIAC under heating at temperatures from 350 to 444°C

Experiment conditions	SIAC-5	SIAC-commercial	SIAC-11	VAC
Before Hg adsorption	1.4	3.7	2.3	1.4
After Hg adsorption	1.6	1.4	2.3	1.2
After SIAC drying and before Hg adsorption	–	5.0	4.0	–

endothermic peak at temperatures around 450°C (Fig. 7) is accompanied by an accelerated mass loss and may be attributed to the evaporation of elemental sulphur because the major form of sulphur in SIAC-commercial is elemental. Figure 8 is for SIAC-commercial after drying and before Hg adsorption. The drying process was carried out in an oven at 105°C and had resulted in a substantial removal of moisture. The cause of the endothermic process associated with a significant mass loss at temperatures close to 450°C was not determined.

The mass loss between 350 and 444°C, which are close to boiling point of mercury and sulphur, was determined (Table 4). The mass loss before Hg adsorption was 1.4% for SIAC-5 and 3.7% for SIAC-commercial. Incidentally, the sulphur content

in SIAC-5 was lower, 5.1% (Table 1). After Hg adsorption, the mass loss varies from 1.4% for SIAC-commercial to 2.3% for SIAC-11. It is expected that the sulfur-free AC had the smallest mass loss, 1.2%. The mass loss for all SIAC in the temperature range of 350–444°C (Table 4) was much smaller than the total sulphur content (Table 1), suggesting other forms of sulphur in SIACs. It is noted that after drying the mass loss between 350 and 444°C increased from 3.7 to 5.0% for SIAC-commercial (Table 4 and Fig. 8) and from 2.3 to 4.0% for SIAC-11 (Table 4 and Fig. 9).

Although boiling point of mercury is 356.7°C, the mass loss after Hg adsorption may not be attributed to the evaporation of mercury since the amount of Hg in the spent SIAC was very small (<0.04%, Table 3) and the form of Hg in the spent SIAC is unlikely elemental Hg. It is evident that Hg adsorption by SIAC is a process dominated by chemical processes, particularly at relatively high temperatures over 75°C.

Calorimetric analysis

The enthalpy values of Hg-SIAC interaction can provide information about mechanisms of Hg adsorption. Understanding the mechanisms of Hg adsorption is essential to optimizing activated carbons for better Hg removal efficiency and to developing technologies for handling of the spent AC. Thus, the enthalpy profile, which is the enthalpy of the adsorbed phase as a function of loading, can help characterize the adsorbents and learn their structure [33, 34]. For example, Myers *et al.* used the energy profile to predict multicomponent adsorption equilibrium for five gases on NaX [34].

In this study, the integral heats of liquid mercury interaction with activated carbons, sulphurized and not, were determined at three temperatures and at different Hg/AC mass ratio. The Hg/AC mass ratio was changed by varying the amount of Hg. The results showed that the interaction between AC and Hg is a complicated process. The overall process can be either exothermic or endothermic with enthalpy values ranging from -3.6 to 1.9 J mol^{-1} (Table 5).

During contact of liquid Hg with porous AC, several processes may occur including exothermic physical adsorption of Hg on carbon, the formation of multiple layers of Hg on carbon surface, and lateral adsorbate-adsorbate interactions that both are

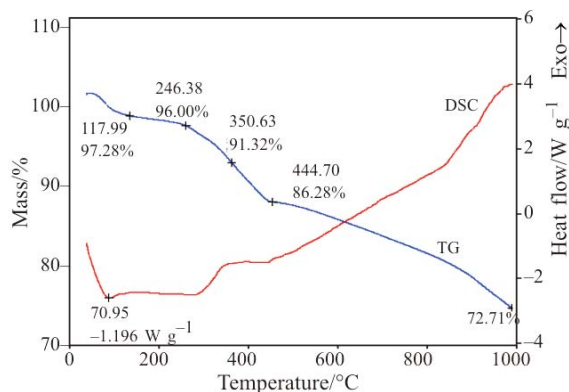
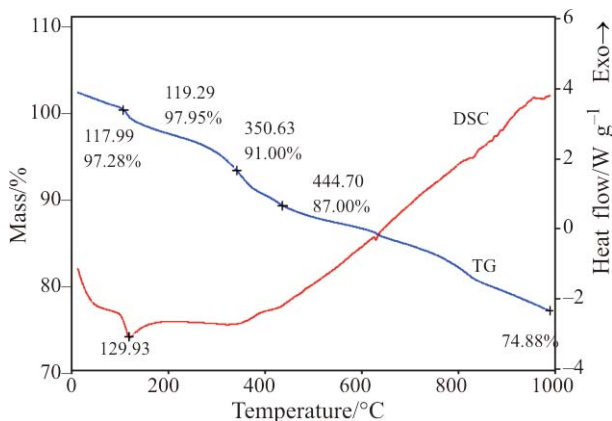

Fig. 8 DSC and TG curves of SIAC commercial after drying and before Hg adsorption

Fig. 9 DSC and TG curves of SIAC-11 after drying and before Hg adsorption

Table 5 Enthalpy of liquid mercury interaction with AC at different Hg/AC mass ratio and temperature

Type of AC	Temperature/°C	Hg/AC mass ratio	Enthalpy $\Delta H/\text{J mol}^{-1}$	Equilibrium time/min
AC-KOH	30	0.25	-965	9
		0.50	-12	4
		1.23	-24	11
		1.92	77	20
		2.26	67	34
	40	0.25	1206	30
		0.74	635	32
		0.95	475	40
		1.23	372	43
		2.00	95	39
		2.70	96	49
	50	0.25	1914	31
		0.50	1354	80
		0.75	980	44
		1.24	622	55
1.50		517	57	
2.00		359	40	
2.20		250	50	
3.00		239	43	
SIAC commercial	30	0.50	-78	10
		0.75	-7	5
		1.74	-14	9
		2.00	-11	12
		2.50	-4	8
SIAC-SO ₂	30	0.25	289	37
		0.74	112	28
		1.50	25	20
		2.50	-17	10

endothermic, the endothermic pore diffusion of Hg and chemical adsorption of Hg, which is likely an endothermic process. Therefore, the sign of the overall enthalpy is a qualitative measure of the extent of the processes taking place. A large positive (endothermic) enthalpy may be an indicator of predominantly chemical adsorption, while an overall exothermic process suggests a largely physical process. As indicated by data in Table 5, negative values were obtained only at 30°C. All the experiments at 40 and 50°C had positive enthalpy. This is consistent with the fact that chemical adsorption tend to dominate at higher temperatures. Goel *et al.* reported that increased temperature resulted in increased Hg(II) uptake from aqueous solution [35]. Similar results were reported for the Hg(II) uptake by

zeolite [36] and by activated carbons produced from bagasse pith [37].

There was also difference in the thermal behaviour at 30°C between SIAC-commercial and SIAC-SO₂ (Fig. 10); SIAC-commercial constantly gave negative enthalpies while the values of enthalpy for SIAC-SO₂ were always positive except when the Hg/AC mass ratio was at 2.5. SIAC-SO₂ has a surface area only 40 m² g⁻¹ which is less than a tenth of that of SIAC-commercial (660 m² g⁻¹). Another difference is that SIAC-SO₂ has higher sulphur content (10.2%) than SIAC-commercial (8.1%). Apparently, for SIAC-SO₂ physical adsorption of Hg is much less important than for SIAC-commercial. For physical adsorption to be effective, specific surface area of an activated carbon must be high.

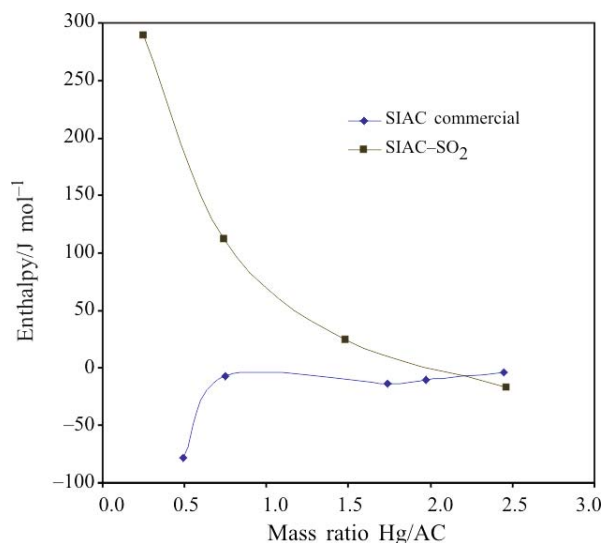


Fig. 10 Enthalpy of mercury interaction with SIAC-commercial and SIAC-SO₂ vs. Hg/AC mass ratio at 30°C

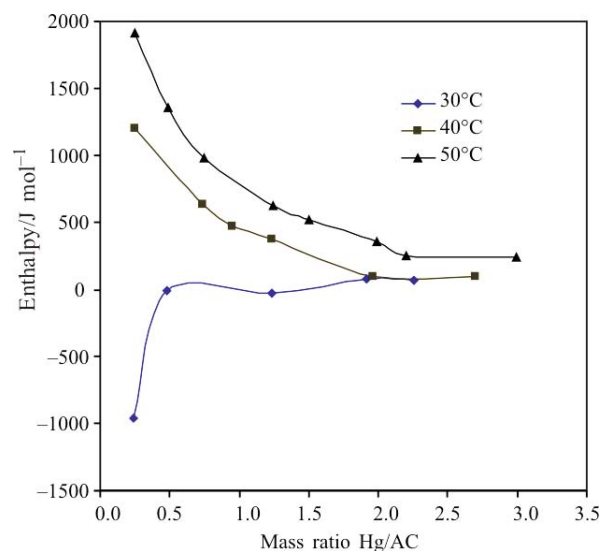


Fig. 11 Enthalpy of mercury interaction with AC-KOH vs. Hg/AC mass ratio at 30, 40 and 50°C

When mercury adsorbed on AC-KOH at 30°C, the overall process was also exothermic or weakly endothermic (Table 5). AC-KOH was highly porous with a specific surface area of 1930 m² g⁻¹ and low sulphur content. With AC-KOH, the overall process became endothermic with rising temperature from 30 to 50°C (Fig. 11), suggesting a shift from physical to chemical process. As shown in Table 5, in general, the equilibrium time increased with temperature, which might suggest that the chemical process was slower than the physical process. The chemisorption of Hg on AC (both sulphurized and not), most likely, occurred due to the reaction of mercury with oxygen and sulphur containing surface functional groups on carbon surface

[28, 38]. It was found that lactone and carbonyl groups are the likely active sites for elemental mercury adsorption which follow a reduction–oxidation mechanism; that the phenol groups present in carbon may inhibit mercury adsorption [28]. Yan *et al.* reported that for sulphur-impregnated carbons, adsorption of elemental mercury involves both physical and chemical processes [38].

It is interesting to note that the effects of Hg/AC mass ratio on the value of enthalpy and on equilibrium time were rather complicated. For example, depending on temperature, in the case of AC-KOH (Fig. 11) the value of enthalpy could increase or decrease with Hg/AC mass ratio. Moreover, there seemed a positive dependence of equilibrium time on the Hg/AC mass ratio for AC-KOH at 30°C, while the dependence was negative for SIAC-SO₂ (Table 5). Although a positive dependence on Hg/AC mass ratio might suggest that the supply of Hg is a rate-limiting step, a clear elucidation of mechanisms behind these observations is not feasible at this stage.

Conclusions

Thermal analysis of SIAC before and after mercury uptake from vapour phase showed that DSC curves suggest both endothermic and exothermic changes during heating. The endothermic processes were attributed to evaporation of moisture and other volatile components. The exothermic processes existed in a wide temperature range, 150–850°C, likely due to the thermal decomposition of oxygen-containing surface functional groups and, as a result, outgassing of carbon oxides (CO and CO₂). Due to the low content of mercury in spent SIAC (<0.04%) and high total mass loss of samples under heating (up to 32%), the analysis was not conclusive as to the fate of absorbed mercury during heating. However, the chemical nature of Hg sorption by SIAC at relatively high temperatures is confirmed by the calorimetric study. Depending on the type of carbons, temperature (30 to 50°C) and Hg/AC mass ratio (0.25–3.00), the overall enthalpy of the interaction of liquid mercury with SIAC varied from –3.6 to +1.9 kJ mol⁻¹, suggesting both physical and/or chemical adsorption. With the increase in temperature, there seemed a shift from the exothermic physical adsorption to the chemisorption that is often endothermic. Physical adsorption contributes substantially to the mercury adsorption on activated carbons with relatively higher specific surface area and lower sulphur content, particularly at lower temperatures. Chemical adsorption of mercury seems to dominate for activated carbons with relatively lower specific surface area and higher sulphur content at higher temperatures.

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